Analysis of coloured Grooved Ware sherds from the Ness of Brodgar, Orkney

Manuscript Details

Manuscript number	JASREP_2019_293
Title	Analysis of coloured Grooved Ware sherds from the Ness of Brodgar, Orkney
Short title	Analysis of coloured Grooved Ware sherds from the Ness of Brodgar, Orkney
Article type	Research Paper

Abstract

To the accumulation of evidence of painted decoration applied to Neolithic and Early Bronze Age pottery in Britain and elsewhere in Europe we report here the presence of decoration in red, black and white on some third millennium BC Grooved ware pottery at the Ness of Brodgar on Orkney. As expected, the red was identified as an iron-rich material and black was carbon black. The white was prepared from calcined (cow) bone; however, its identification encountered some issues arising principally from the effects of the prevailing burial conditions on the bone. Furthermore, whereas the chemical and FTIR data were consistent with the presence of apatite, XRD indicated that the white had a significant content of silicate minerals. This finding has suggested that the white required a preparatory step which might have included the calcined bone bring ground to powder in a stone mortar before application to the vessel surface. The results obtained at Ness of Brodgar are reviewed in the light of archaeometric data on similarly decorated prehistoric pottery reported from sites in Europe.

Keywords	Neolithic pottery; Ness of Brodgar Orkney; painted decoration; SEM-EDX; pXRF; XRD; FTIR
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Submission Files Included in this PDF

File Name [File Type]

Letter to Editor.pdf [Cover Letter]

Ness June 10.doc [Manuscript File]

Table 2a Compositions determined by pXRF.docx [Table]

Table 2b bone pXRF.docx [Table]

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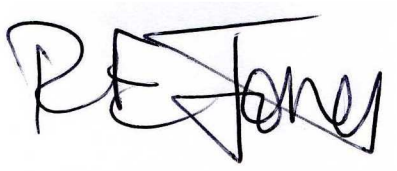
June 12, 2019

Editor in Chief
Journal of Archaeological Science Reports

Dear Editor

I am submitting the paper by myself, Card, Towers and Odling, entitled ***Analysis of coloured Grooved Ware sherds from the Ness of Brodgar, Orkney***. Presenting the results of analysis of decorated Neolithic sherds from Orkney's premier Neolithic site, this paper documents the earliest instance of deliberate decoration of pottery in the UK. The three main colourants have been identified through the application of several techniques and are discussed in relation to the corresponding occurrence of decoration in prehistoric ceramics elsewhere in the UK and on the continent. We believe the paper is an important contribution to the current interest in an 'archaeology of the senses' and more particularly the presence and meaning of colour in prehistory. We are pleased to submit our paper to *JAS Reports* in part because *JAS* has already published papers (fully acknowledged in our paper) on similarly decorated pottery.

Yours sincerely



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Analysis of coloured Grooved Ware sherds from the Ness of Brodgar, Orkney

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Abstract

To the accumulation of evidence of painted decoration applied to Neolithic and Early Bronze Age pottery in Britain and elsewhere in Europe we report here the presence of decoration in red, black and white on some third millennium BC Grooved ware pottery at the Ness of Brodgar on Orkney. As expected, the red was identified as an iron-rich material and black was carbon black. The white was prepared from calcined (cow) bone; however, its identification encountered some issues arising principally from the effects of the prevailing burial conditions on the bone. Furthermore, whereas the chemical and FTIR data were consistent with the presence of apatite, XRD indicated that the white had a significant content of silicate minerals. This finding has suggested that the white required a preparatory step which might have included the calcined bone bring ground to powder in a stone mortar before application to the vessel surface. The results obtained at Ness of Brodgar are reviewed in the light of archaeometric data on similarly decorated prehistoric pottery reported from sites in Europe.

Keywords: Grooved ware pottery, Neolithic, Ness of Brodgar, Orkney, paint, bone white, SEM-EDX, pXRF, XRD, FTIR

1. Introduction

Since the identification of Grooved Ware pottery as a distinct pottery tradition (Piggott, 1936), its geographical range and its various physical forms have been hugely extended (e.g. Grogan and Roche 2010.). Nowhere is this more apparent than in Orkney where several 'classic' Grooved Ware sites have been excavated: Skara Brae (Clarke and Shepherd, forthcoming), Rinyo (Childe and Grant 1939), Barnhouse (Richards, 2005), Pool (Hunter et al., 2007), Links of Noltland (Moore and Wilson, 2011), Crossiecrown (Card et al 2016), Stones of Stenness (Ritchie, 1976), Quanterness (Renfrew, 1979). To this catalogue can now be added the Ness of Brodgar (Card et al., 2017; Card, 2018; Towers et al., 2017) where not only has a vast range of Grooved Ware been recovered but new decorative techniques utilising colour have been recognised.

During the 2011 season of Ness excavations it was suspected that a few sherds showed signs of having coloured decoration. This aroused considerable interest as colour, both applied and a variety of naturally occurring coloured sandstones had been incorporated into built structures, had already been noted at the site. As the ceramic material was examined in greater detail as it emerged from the ground, it became clear that a number of sherds had a coloured layer deliberately applied to their exterior surfaces. The colours involved were red, white and black.

The Grooved Ware sherds were decorated with plain applied cordons and the colours appeared to have been applied differentially. The red colouring was identified solely on the plain cordons (SF 10930) and the black colouring had been applied, either up to the edge of a cordon, or up to and including a cordon (SF 10541). In further contrast, the white colouring was often applied as a thick, all-over slip.

The aim of this paper is to report on the material characterisation of the three main colourants on the Grooved ware sherds at Ness of Brodgar using a variety of analytical techniques. Examples of animal bone from the same contexts as the pottery at Ness were similarly analysed. The results are discussed and compared with those obtained on similarly decorated early prehistoric pottery from several European contexts. However due to the ongoing nature of the excavation, the post-excavation programme and the complexity of the stratigraphic narrative of the Ness, it is presently not possible to attribute confidently the definitive context of all the coloured pottery.

2. Material

Table 1 introduces the 23 sherds examined, some of which are illustrated in Fig. 1. The number of examples examined of each colourant is uneven; more examples of white were

selected reflecting the issues arising from their characterisation. Cow bones were selected from contexts 2275, 4000 and 4010 at Ness of Brodgar: seven were calcined with a white colour, five were unburnt (usually cream coloured) and five were carbonised.

Small Find number	*Context*	*Structure*	*Description and colour*
860	1001	Lower ploughsoil	Ephemeral white on rim and cordon
861	1001	Lower ploughsoil	Ephemeral white
882	1007	Midden around St 1	Fugitive white
1518	1109	St 1 infill	Ephemeral white
6381	2817	Late midden to N of St 12	chalky white
8509	2275	St 12 infill	White
8509b	2275	St 12 infill	White
9871	2306	St 12 annex infill	white
10252	2306	St 12 annex finds deposit	white
10533	2306	St 12 annex finds deposit	White (speckled with charcoal) on cordon
10930b	2315	12 annex	White on cordon and body
10938	2315	12 annex	Thin white on cordon
10982	2315	12 annex	Distinct white layer on cordon
11247	4000	St 12 infill	White
12018	4010	St 12 infill	White
12631	4017	St 12	White
27437	6650	Tr T midden	White
14463	4033	St 12 infill	Black and white
6798	2833	St 12 infill	Black applied to well smoothed surface up to two of the distinctive set of three converging cordons; fugitive white in cordon groove(s); possible red

10541	3054	Central Midden Area	Black applied to cordons and area between two converging cordons
9486	2312	St 12 infill	Thin pink/red wash on cordon of a large vessel
10101	2278	St 12 annex infill	Red wash on cordon
10930	2315	12 annex	Red pigmented band running along the upper half of the applied cordon

68 Table 1 The decorated sherds from Ness of Brodgar.

69

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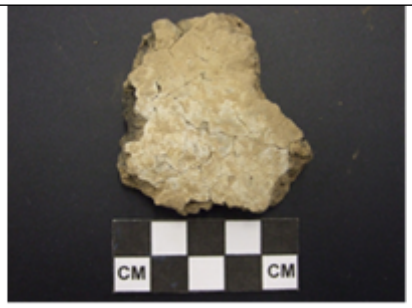
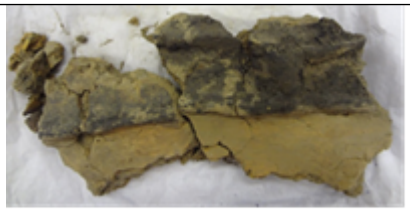
SF			
8509		*6798*	
10930b		*10541*	
10938		*9486*	

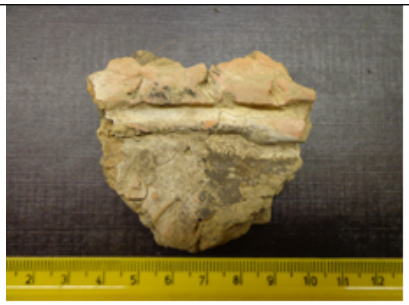
14463		*10930*	

Fig. 1 Examples of decorated sherds from Ness of Brodgar. © ORCA

3. Methods

Owing to their special status together with the sometimes fugitive nature of the coloured surface and frequent friable fabric, the sherds had necessarily received minimal cleaning following excavation. As a result, the preparation of samples for analysis was in a few instances problematic; the issue of soil or other contamination is mentioned below and is taken up in the Discussion.

The sherds were examined with a Leica LED3000 stereomicroscope. Elemental analyses were carried out (a) non-destructively by portable X-ray fluorescence (pXRF) with a Niton XL3t 900 SHE GOLDD Alloy Analyser, with a 50kV Ag X-ray tube, 80MHz real time digital signal processing and two processors for computation and data storage respectively; analyses were undertaken in the TestAllGeo calibration within the Soils and Minerals mode with resolution of *c.*165 eV at 35 KeV; the analysis area was *c.* 8mm^2, (b) an EDAX Oxford Microanalysis system attached to a Zeiss Sigma field-emission analytical SEM at Glasgow University Geosciences ISAAC facility and (c) a Hitachi S-2700 SEM with an Oxford Inca 350 analyser at the Advanced Materials Research Laboratory at Strathclyde University. Analysis and imaging in (b) was of the surface (viewed as a secondary emission (SE) image) and in (c) of a *section* (viewed in back scattered mode) revealing the fabric, interface of body with surface decoration, and decorated surface; area analyses (150 μ^2 in the case of c) at different positions were made, giving (normalised) concentrations of twelve and eleven elements respectively. As regards preparation for (c), the cut fragment was impregnated and embedded in resin, then polished and gold coated prior to examination. The combination of a poorly fired fabric and the frequently thin, poorly adhering decorated surface seriously limited the success of preparing resin-embedded samples.

For FTIR (Shimadzu FTIR 8400 S) and powder X-ray diffraction (Bruker D8 Advance using Cu Kα radiation with Sol-X Energy Dispersive detector and TOPAS 3.0 Rietveld analysis software) small scrapings were taken from the surface with as little of the underlying body as possible; additional

samples of the body fabric were prepared separately and were then finely ground in an agate mortar for XRD analysis. The bone was cleaned of any visible soil or other accretion before a small fragment was crushed to powder. Raman spectra were obtained with a Renishaw inVia laser Raman microscope operating with a 785 nm laser. Thin sections prepared from some undecorated sherds from the same contexts as those with colouration were examined petrographically with a Leica Wild M240 polarising microscope.

4. Results

4.1 General observations

The fabric making up the body of all the sherds was coarse, usually abraded, and commonly comprising up to 50% large angular rock fragment temper. Among the large inclusions, ranging in size up to 1 cm, siltstone was the most frequently observed in thin section, followed by sandstone and igneous dyke material, all of them familiar from the corresponding Grooved ware examined petrographically at the neighbouring settlement of Barnhouse (Jones, 2005). More unusual were examples with a less coarse fabric probably tempered with shell (e.g. SF 10982), also observed at Barnhouse. The applied cordons were prepared from a finer clay.

The pottery had been poorly, and usually unevenly fired leaving a dark reduced core; many of the sherds were very friable. Their thickness ranged from *c*. 7 to 18 mm. Sooting or carbonised residue was evident on the interior surface of several sherds, this surface being lightly smoothed, but sometimes not at all. By comparison, the exterior surfaces, usually light coloured and smoother, had probably been prepared with considerably greater care.

To the comment above about the locations of the colourants on the exterior surface should be added the observation that white seems to have been applied equally to cordons and to the plain surface. The morphology of the decorated layer varied considerably according to its thickness and level of attrition: the reds were generally in the form of a thin, dilute ‘wash’ (Fig. 2a), much of which may well have been absorbed into the fabric on application; black by contrast was more coherent, adhering quite well to the surface (Fig. 2b), and white ranged from a distinct secure layer (in the case of SGF 10930b with a blueish tinge in places (Fig. 2c)) to a thin sometimes fugitive slip (Fig. 2d) to one that was almost ephemeral and pale coloured. Small charcoal fragments adhering to the white were evident in SF 10533. SF 6798 was the only example in which possible red appeared alongside black and white in the cordon. The white in SF 14463 underlies the black layer (Fig. 2e).

The quality of the surface on to which the white was applied, whether the fine clay of the cordon or the coarse clay of the body, did not apparently affect the extent of adherence of the paint/slip.

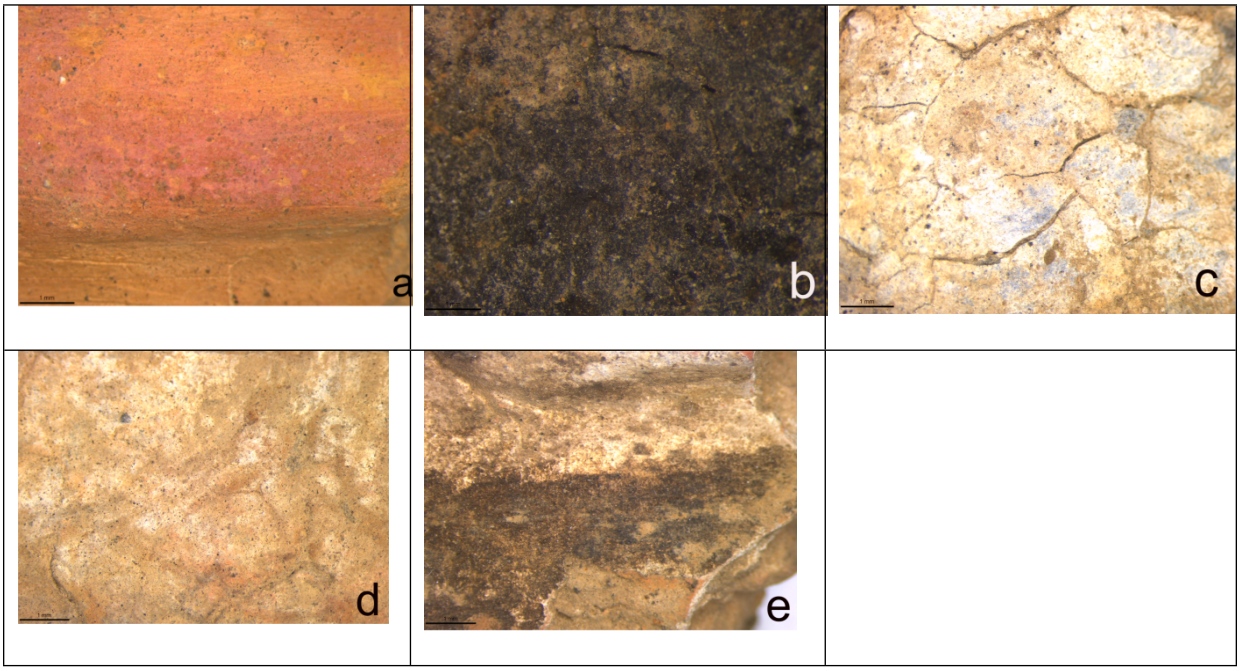


Fig. 2 (a) Red band on SF 10930 contrasting with the orange-red of the fabric, (b) black on SF 10541, (c) white on SF 10930b, (d) fugitive white on SF 12018 and (e) black overlying white in SF 14463. The 1mm scale appears bottom left in all images.

4.2 White

The sherds with white decoration were first analysed by pXRF: on the decorated surface, the interior surface (which was sometimes coated with carbonised residue) and the fabric. Variations are observed in many element concentrations among the sherds and between the different locations on each sherd (Table 2a); much of this variation should be attributed to the coarse nature of the fabric. However, it is clear that the white decoration on most sherds is richer in calcium (Ca) and phosphorus (P) than the body material (Fig. 3), a feature characteristic of bone ash. Nevertheless, the Ca and P contents in the white appear to form three groups:

A. high Ca (3-7%) and P (8-10%): SF 8509, 10930, 10938, 11247, 14463 and 27.437

B. medium Ca (1.5-4%) and P (3-5%): SF 882, 9871, 1109, 10252, 10982, 12018 and 14463

C. low Ca (<1%) and P (<2%): SF 860, 861 and 6381

Also plotted on Fig. 3 are the results for the bone which show a significant range of Ca and P contents, the calcined and the unburnt bone having the highest and lowest contents respectively. Table 2b presents the individual bone compositions.

Looking at the results more fully, lying outside Group A but with low Ca are the interior surface of SF 8509b and the body of SF 10938 which both contain high P (10%). Lying between Group B, which comprises only examples of white decorated surface, and Group A are three instances of interior surfaces with high P (SF 10533, 11247 and 12018). The carbonised and possibly soil-concreted interior surface of SF 11247 is likely to be responsible for the high P; its high Ca could relate to the presence in the fabric of small white inclusions which may be shell. Into Group C fall SF 860, 861 and 6381 and the remaining body and interior surfaces. The absence of a significant contrast in composition between exterior surface and body of these three sherds strongly suggests that these elements, or indeed any other elements, were not responsible for the pale or white-coloured surface. In the case of the rim sherd, SF 860, the pale colouration is likely to be the result of firing; SF 861 is a pale probably unfired clay, and SF 6381 appears to be an amorphous accretion of pale clay to a dark fragment. SF 9871 (like SF 9486) has a higher Ca in the dark carbonised residue on the interior surface than in the body and differs from the body in most other elements.

The explanation of the difference between Groups A and B may be the thickness and 'density' of the white layer. It is apparent, for example, that the decorated area on SF 10982 is more like a slip than a painted area.

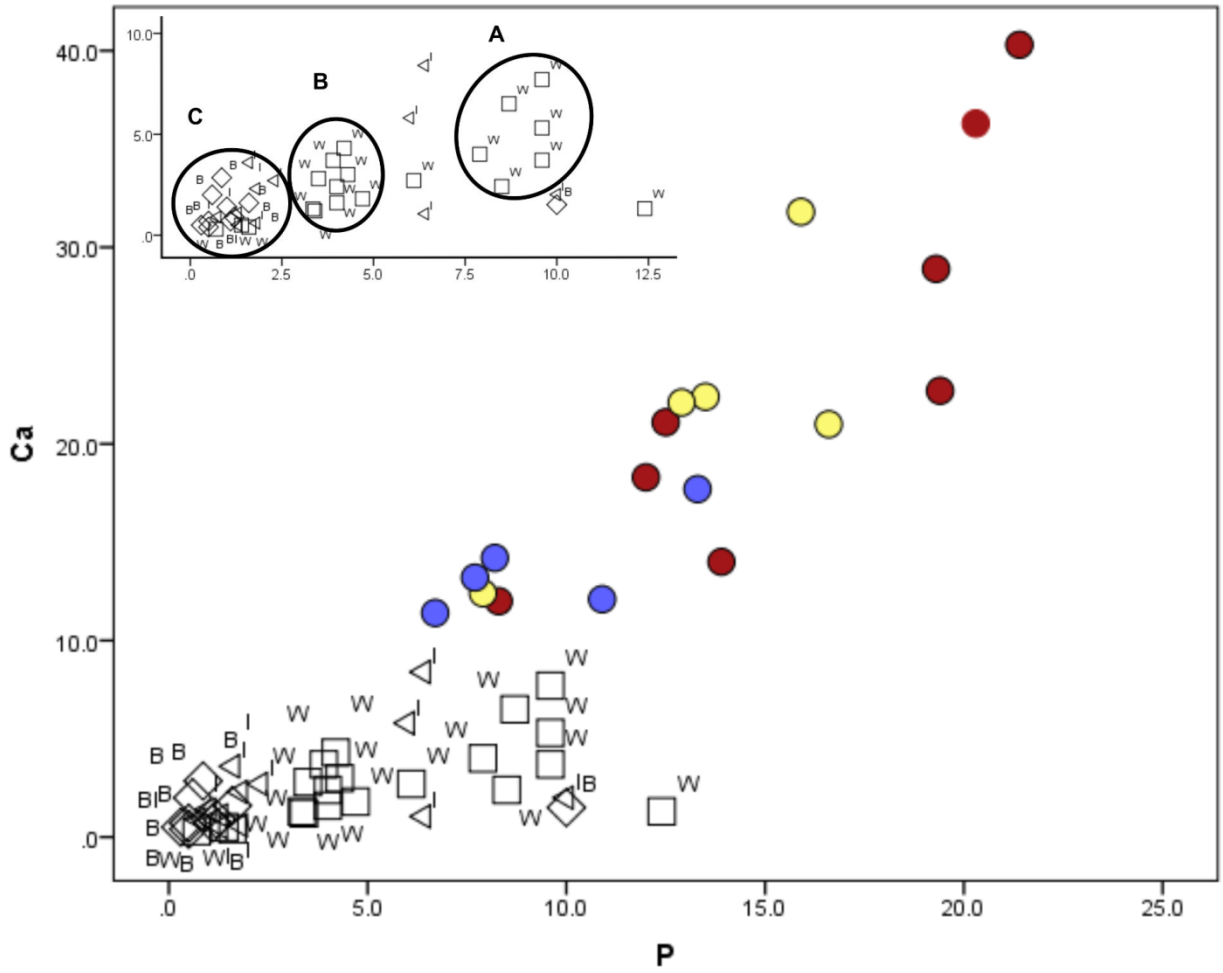


Fig. 3 Ca-P plot of examples of white decoration (W), interior surface (I) and body (B) in the decorated sherds; pXRF data. Bone: blue unburnt, yellow carbonised, red calcined. The inset shows Groups A, B and C which represent high, medium and low P respectively.

TABLEs 2a and 2b to be inserted here – *currently separate docs*.

The surface of two white decorated sherds – SF 8509 and SF 10930 - and the section of another such sherd – SF 12631 - were examined in the SEM (Figs. 4, 5).

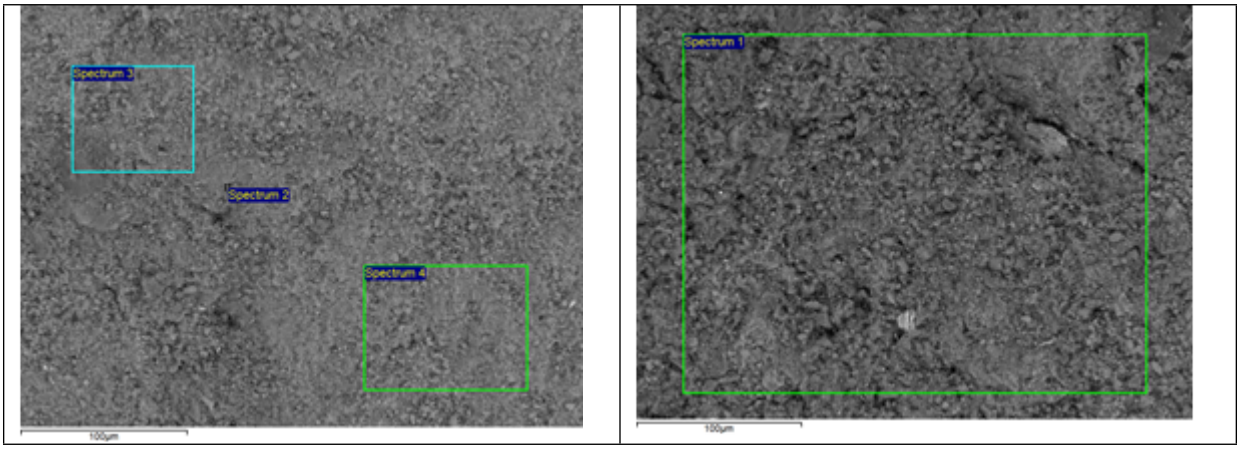


Fig. 4 SEM (SE) images of SF 8509 (a) red layer showing three analysis positions and (b) adjacent undecorated area (single area analysis).

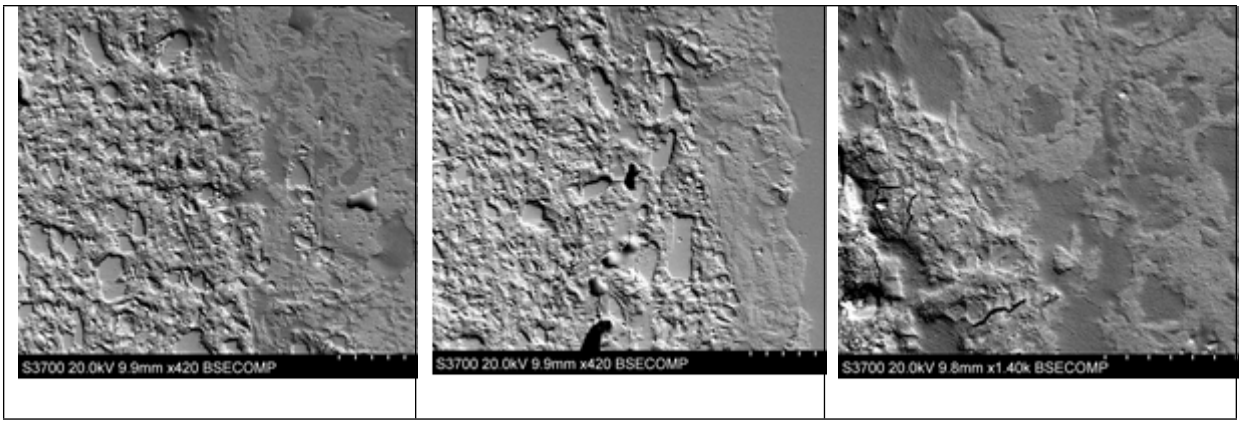


Fig. 5 Back-scattered SEM images of SF 12631: cross-sections through the body and outer coloured layer. (a) and (b) two views of the body (left of image) decorated layer (right of image); (c) interface between surface layer and body at higher magnification.

The white surface of SF 8509 appears smoother and finer textured than the undecorated area (Figs. 4a and b). Analyses of the three decorated spots (Fig. 4a) consistently show higher P and Ca than in the undecorated surface (Table 2a). On the other hand, the corresponding contrast between decorated and undecorated spots on the exterior of SF 10930B is much smaller. The characteristics of the decorated layer on SF 12631 as viewed in Fig. 5 (as back-scattered images) are as follows: the thickness varies up to 50µ; the texture is smooth, fine-grained with occasional to rare large grains; that the material layer is not uniform is evident from the presence of pores of different shapes up to 15µ in size, although it is possible that some of them may have been introduced during the thin section preparation. The demarcation between this layer and the body is clearly visible as if to suggest no reaction between them has taken place. This observation relating to whether decoration

occurred before or after firing is taken up in the discussion. The P and Ca contents in the white surface material are strikingly higher compared to the body. The slightly higher magnesium present in the surface material may be ascribed to this element's natural presence in bone and bone ash.

When analyses were repeated to include the detection of carbon, values of *c.* 40% and 70% carbon were determined in the surface and body respectively of SF 12631. While most of this carbon should be due to the embedding resin, the presence of a small component of carbonate in the bone ash (Roberts *et al* 2008, 326) could account for the remainder as could remnant proteinaceous carbon resulting from incomplete calcination of the original bone, and even, although less likely, organic adhesive. The relatively higher carbon in the body may reflect the presence of that element entering the fabric during firing.

SF Number	O	P	Si	Ca	C	Fe	Al	Na	Mg	K	Mn	Ti	Total
8509 white	50.49	11.39	9.19	8.44	5.28	3.94	3.2	2.95	2.45	1.63	0.8	0.25	100
8509 unpainted ext	52.85	5.89	15.51	4.32	3.8	5.09	5.99	1.67	1.44	2.15	0.69	0.59	100
10930B white	46.71	7.35	12.69	3.44	12.79	5.26	4.95	2.47	1.68	1.96	0.29	0.41	100
10930B unpainted ext	44.17	6.66	12.94	2.38	16.49	5.73	4.3	2.33	1.58	2.58	0.34	0.5	100
12631 white	74.05	8.86	1.55	7.41	n.d.	0	3.37	1.69	2.71	0.35	n.d.	0	100
12631 white	69.52	12.67	0.65	10.49	n.d.	0.65	1.52	1.72	2.78	0	n.d.	0	100
12631 white	69.41	12.53	0.66	10.43	n.d.	0.51	2.11	1.48	2.6	0.26	n.d.	0	100
12631 white	68.44	12	2.41	9.77	n.d.	0.61	2.73	1.05	2.55	0.43	n.d.	0	100
12631 Interface body-white surface	69.31	3.34	13.12	3.11	n.d.	0	7.03	0.86	1.32	1.33	n.d.	0	100
12631 body	64.48	0	21.68	0.4	n.d.	1.39	8.23	1.19	1.19	1.44	n.d.	0	100
12631 body	64.33	0	19.71	0.42	n.d.	0.85	9.62	1.66	1.9	1.34	n.d.	0.17	100
12631 body	62.65	0	21.84	0.44	n.d.	0.87	9.04	2.48	1.2	1.3	n.d.	0.17	100

10541 black	38.63	0.89	3.82	2.37	46.88	2.81	2.8	<0.1	0.3	0.56	0.61	n.d.	100
10541 fabric	49.92	16	15.63	0.56	16.55	4.87	7.36	0.52	0.94	2.08	<0.1	0.57	100
10930 red	64.53	3.03	18.73	3.91	3.71	6.92	6.18	0.47	0.74	5.25	n.d.	d	100
10930 undecorated ext	51.67	3.1	15.08	4.09	8.75	5.03	6.53	0.54	1.02	3.48	n.d.	0.45	100

Table 3 The SEM-EDX determined compositions of decorated and undecorated exterior surface of SF 8509 and SF 10930b, and of surface, interface and body of SF 12631, expressed as wt % element. N.d. not determined.

The principal **FTIR** absorption peak in the white of SF 8509 and 10252 occurs at 1014-1016 cm^{-1} followed by ones at 960-962 cm^{-1} and 559 cm^{-1}, all of them corresponding to the phosphate group in hydroxyapatite; similar results have been obtained by Odriozola and Hutardo Perez (2007, 1799) and Roberts et al. (2008, 326). Comparing the spectra of the white in SF 10252 with those of calcined, unburnt and carbonised bone from Ness of Brodgar clearly indicated a close match with calcined bone (Fig. 6).

Raman analysis of the decorated surface of SF 10252 revealed a very weak broad peak at 955 cm^{-1} close to that of apatite at 961 cm^{-1}. Nothing was detected in SF 11247 and SF 12631.

More informative are the results of mineralogical analysis by **X-ray diffraction** (XRD) carried out in most cases on both the white and the body (Table 4). The surface layer is richer in feldspars, clays and muscovite compared to the body but the apatite content is low, estimated at <1%. As expected, the bone is consistently apatite; there is a low quartz content in the (black) carbonised bone, and uncalcined bone shows a high background consistent with a large non-diffracting fraction.

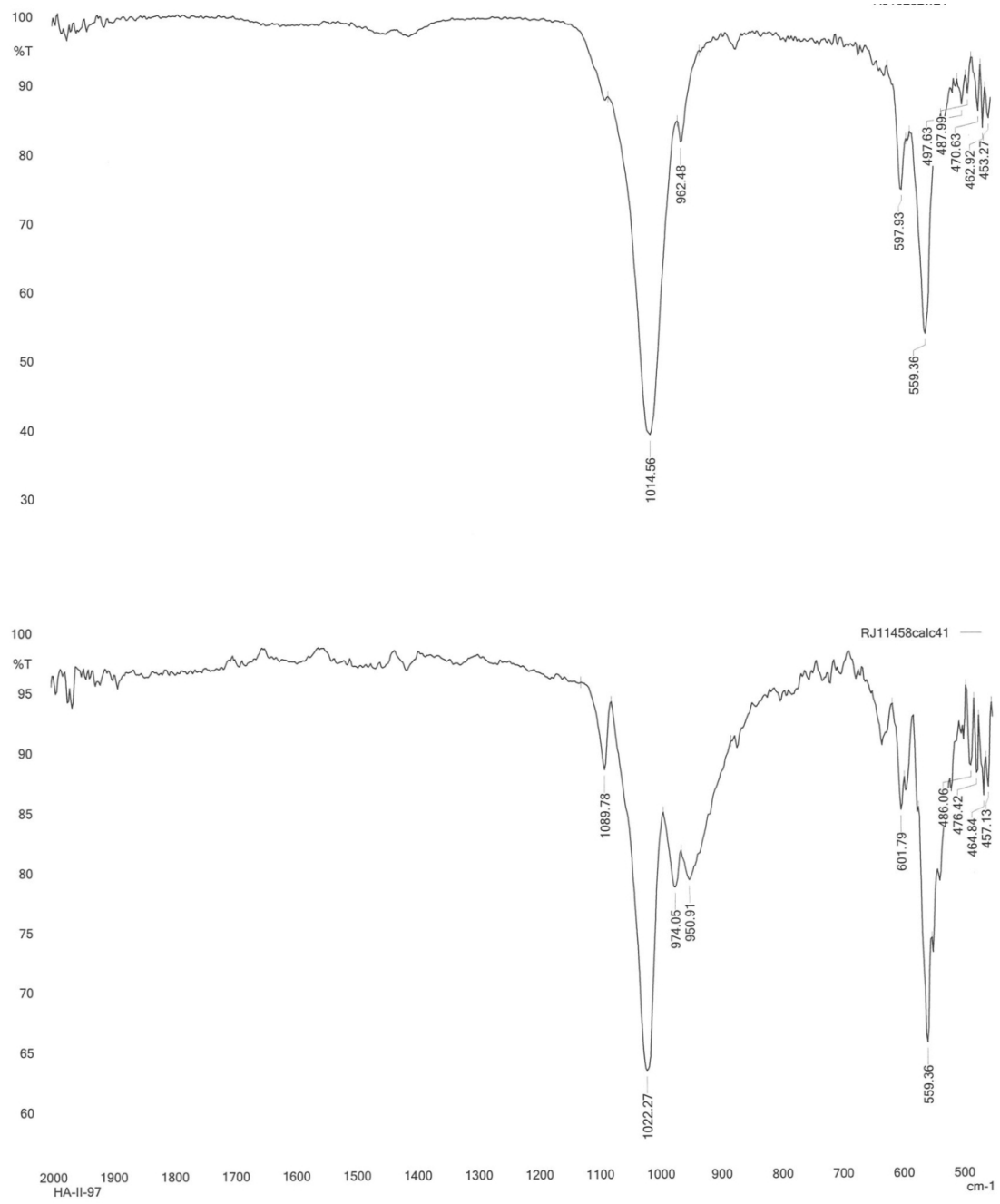


Fig. 6 FTIR spectra in the 500-2000 cm^{-1} range of the white on SF10252 (top) and calcined bone from Ness of Brodgar (bottom).

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Small Find Number	*Quartz*	*Albite* $NaAlSi_3O_8$	*Plagioclase* $NaAlSi_3O_8$ – $CaAl_2Si_2O_8$	*Clay minerals*	*Chlorite*	*K-feldspar* $KAlSi_3O_8$	*Muscovite* $KAl_2(AlSi_3O_{10})(FOH)_2$
9871 surface	25.1		19	9.3	0.5	24.4	14.9
9871 body	36.3	27.7	19		1.8	13.4	7.1
11547 surface	26.1		21.6	6.4	0.8	19.5	9.4
11547 body	43.3	34.8			1.4	7.1	5.5
27347 surface	23.7		31.7	8.1	2.1	16.2	10.3
27437 body	31.8	45.7			1.1	7.8	4.9
10252 surface	25.4		31.0	7.4	1.6	20.6	12.1
10252 body	53.1	28.7			1.4	10.1	3.4
14463 surface	14.0		32	9		36	
14463 body	40.0		23	8		23	
8509 surface	9.0		47.0	12		27	
12631 surface	22.0		26.0	12		33	
Unburnt bone							
Calcined bone	Very low						
Carbonised bone	Very low						

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Small Find Number	*Diospide* $MgCaSi_2O_6$	*Mica*	*Annite mica* $KFe^{32+}AlSi_3O_{10}(OH)_2$	*Larnite* Ca_2SiO_4	*Apatite*	*Merrilite* $Ca_9NaMg(PO_4)_7$	*Nontronite*	*Alluaudite*
9871 surface					0.5			
9871 body	8.3		4.3				1.1	
11547 surface				5.7	0.6	8.1		1.8
11547 body	2		5.1				0.8	
27347 surface				3.2	0.8	1		3
27437 body	5		2.8				0.9	
10252 surface				1.2	0.5	0.3		0
10252 body	1.9		1				0.4	
14463 surface		9						
14463 body		6						
8509 surface		5						
12631 surface		6						
Unburnt bone					100			
Calcined bone					Major			
Carbonised bone					Major			

242 Table 4 Results of XRD analysis of white decoration and body, giving the estimated percentage presence of minerals.

4.3 Black

The Raman spectrum of the black on SF 6798 and SF 10541 revealed peaks characteristic of crystalline carbon at 1346 and 1585 cm^{-1} (Fig. 7). This is most likely to be carbon black. EDX analysis of the black layer indicated the much higher carbon content in the black than in the body (Table 3). Since pXRF does not detect carbon, little difference in composition between the black layer and the interior surface of SF 6798 was detected (Table 2a).

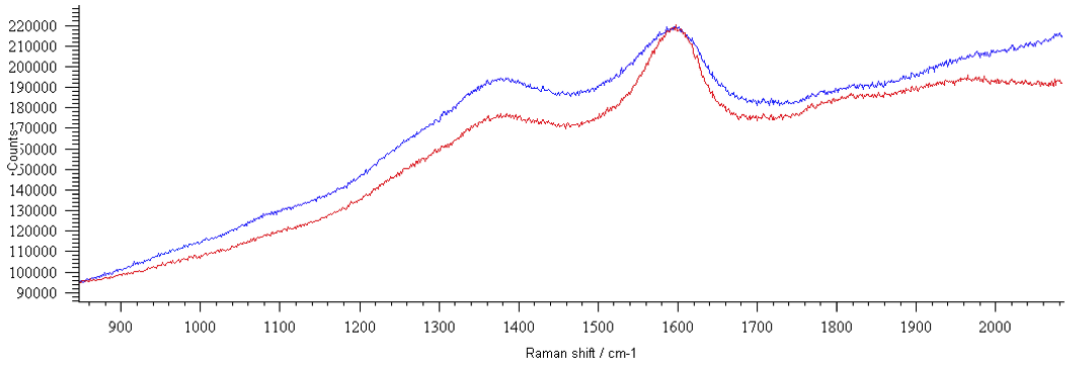

Fig. 7 Raman spectrum of a black spot on SF 10541 (red, bottom) and (modern) coal (blue, top).

4.4 Red

pXRF of SF 10101 and SF 10930 detected higher Fe in the red layer, but this was not the case in SF 9486 (Table 2a). The interior surface of 9486 has a high Ca but low P content, probably due to a calcareous concretion on that surface. The opposite is found in SF 10101 whose interior has a residue with P and low Ca. SEM-EDX analysis of SF 10930 concurs with pXRF in finding a slightly higher Fe content in the red (Figs. 1 and 2a) compared to the body or undecorated exterior surface (Table 3). The morphological contrast between decorated and undecorated surfaces is apparent in the SEM images (Fig. 8).

An important observation from the EDX and pXRF analyses of SF 10930 is the relatively high content of P and Ca in the *undecorated* (exterior) surface, a result which was confirmed by further analysis: 5.1% Ca and 3.9% P in the red and 8.0% Ca and 4.9%P in the undecorated spots. Since XRF analysis of the *interior* surface yields a value of 1% P which lies within the range of what may reasonably be expected in Orcadian clays, one implication is that the surface was coated, deliberately or otherwise, with some bone ash before the red was applied despite the fact that no white is visible today.

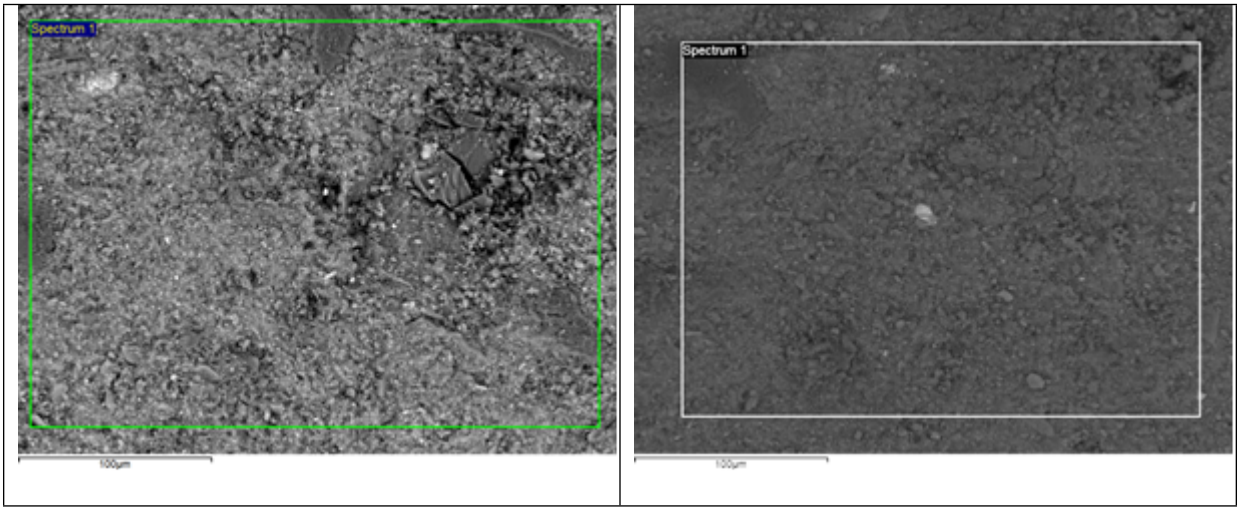

Fig. 8 SEM (SE) images of (a) red decorated and (b) undecorated exterior surface of SF 10930. The areas defined for analysis are marked.

5. Discussion

This discussion can begin with the general observation that the decorated layer, whatever its colour, on the Grooved ware pottery is usually thin, having the appearance of a coloured slip or wash; in only a few cases is it thicker and more coherent. The results for the red and black seem quite straightforward, haematite and carbon respectively being most likely to be responsible for those colours. Considering the former in more detail, the high iron content indicates use of haematite mineral itself (which occurs naturally on the island of Hoy and also on at least one beach in the East Mainland of Orkney (Photos-Jones et al. 2015, 482-84)) or possibly a haematite-enriched clay. In the light of experiments arising from study of the Neolithic haematite finds from Crossiecrown reported by Photos-Jones *et al* (2015), colourant prepared from a lump of haematite mineral by the wet abrasion method could be applied directly to the vessel surface, although it was found to be fugitive. Preparing the colourant within a viscous medium such as gum or resin was found to increase the fineness of the haematite (Isbister in Photos-Jones et al. 2015) and thus in principle its effectiveness as a pigment. This experimental data would seem to harmonise with the evidence of the red on the sherds examined here, that is, their fugitive nature. More stable is the carbon black which was capable of being applied uniformly and coherently to the smoothed surface of the vessel and, at least in one instance (SF 14463), over a layer of white.

298 The presence of high levels of calcium and phosphorus in the pXRF analysis of the white pigment
299 (Fig. 3) coupled with FTIR evidence (Fig. 6) for the presence of phosphate compound(s) strongly
300 supports the conclusion that the white pigment is mainly composed of bone ash. However, although
301 XRD shows that hydroxyapatite, the main mineral component of bone, is present in the bone
302 material recovered from the Ness of Brodgar, there is no evidence for its presence in the white
303 pigment material. Instead, XRD shows that the crystalline components present in the white pigment
304 are quartz, feldspars, clays and other minor silicate minerals. This mineral assemblage might suggest
305 that the white pigment is the use of a white 'clay' material such as has been reported as occurring on
306 the south coast of Orkney mainland (Jones and Brown, 2000). However, the chemical composition
307 of this, or any mixture of the minerals reported by XRD, is severely at odds with the high levels of
308 calcium and phosphorus as reported by pXRF. There are several possible explanations for this
309 conundrum. First, is the possibility that, because of the very thin nature of the white layer, samples
310 taken for XRD analysis were severely contaminated by the underlying body material to an extent
311 that the apatite signal was lost. This is unlikely, although it is relevant to note here that the positive
312 identification, by XRD, of apatite by Odriozola and Hutardo Perez (2007) and Roberts et al. (2008)
313 might be attributed to the thicker, denser and higher fired fabric of the decorative material of the 3rd
314 millennium BC Spanish and Middle Bronze Age Danubian pottery, by comparison with the Ness
315 material. A second, more likely explanation is that the majority of the calcium and phosphorus
316 present in the white pigment is hosted in amorphous material(s) rather than as a crystalline mineral
317 and thus does not respond to XRD spectroscopy. It is widely recognised that the shallow, wet and
318 oxidising burial conditions typical of the Ness locality, promote microbial and fungal degredation of
319 bone material (Turner-Walker, 2008; Jans, 2008). The finely ground nature of the white pigment will
320 make it particularly sensitive to this process, whereas the more massive bone fragments will survive,
321 albeit with evidence of microbial attack on the microscopic scale. It is to be noted that, although
322 microbial attack will denature bone, it does not lead to complete loss of Ca and P from the materials.
323 In support of this, the Ca/P *ratios* of the white pigment samples are close to those of the bone
324 material, the absolute values being lower due to some Ca and P loss coupled with dilution by the
325 presence of silicate minerals. This explains why the Ca/P ratios of <1 observed in the pXRF and SEM-
326 EDX data in Tables 2 and 3 respectively lie well below the ratios of 1.5 to >2.1 found in pottery
327 excavated at drier sites in southern Spain (Odriozola and Hutardo Perez 2007).
328
329 If it is accepted that the P and Ca are evidence of the original presence of bone material in the white
330 pigment, it is possible to calculate the minimum amounts necessary to produce the observed Ca and P
331 levels. Since hydroxyapatite, the mineral constituent of bone, contains 40% Ca and 18.5% P, the 8.9-

12.7% range of P in the white pigment in SFs 8509 and 12631 requires an original hydroxyapatite range of 60-70%. In this light, the substantial amounts of silicate minerals reported from the XRD represent a relatively small component of non-bone material. It is possible that this material was accidentally included during grinding of the bone material in a stone mortar, but this must remain a speculation to be tested by further work.

Those same taphonomic conditions will have also affected the fabric, and most relevant here is phosphorus which is likely to be unevenly distributed as phosphate, in either organic or inorganic forms, in the soil at Ness. Some phosphate will have been absorbed into the pottery fabric at Ness, especially in those contexts at the site where cow bone has been found in remarkably high concentrations (e.g. Mainland et al., 2013). Indeed, the view that the presence of phosphorus within the pottery fabric is due to natural depositional effects is supported by the pXRF analyses which consistently detected both phosphorus and calcium in the fabric, albeit at lower concentration than on the decorated surface (Fig. 3). With one exception (SF 12631), which has not yet been explained, the numerically more limited SEM-EDX analyses also found significant levels of phosphorus in the fabric. But an additional, rather than an alternative, factor is that the co-presence of phosphorus and calcium in the fabric could be attributed to a small amount of bone ash either deliberately added to the clay as part of the potter's 'recipe' or accidentally present. The concept of potters' 'recipes' in Late Neolithic Grooved ware is now well established, observed at Barnhouse in the form of tempering practices (different igneous rock types as well as sandstone, siltstone, mudstone and shell), apparently correlating with particular groups of houses (Jones 2002, 123-131) and to a lesser extent at contemporary settlement sites elsewhere on Mainland Orkney (Jones et al. 2016, 314-5, 345). The white decorated vessels were presumably not used for cooking but even if they had undergone heating on a hearth the resultant temperature effects would have been too low to have affected the morphology/crystallinity of the apatite.

The present study did not specifically investigate the issue of how and when the decoration was applied, however the likelihood is that it was a post-firing process. At the macroscopic level, the observation that the areas of decoration on the sherds lacked signs of a variegated surface and/or smudged carbon is crucial since these two effects, which were common outcomes in the experimental pottery firings carried out on Orkney (Jones et al. 2016, 379, Fig. 11.5.5), would have seriously affected the decoration had it been applied *before* firing. What blemishes there might have been on the vessel surface after firing could have been at least partially erased by 'cleaning' or rubbing the surface before application of the colourant. Unfortunately, the absence of a reaction zone between the body fabric and coloured surface as observed in the SEM (Fig. 5) precludes its use as an indicator of post-firing decoration because the pottery reached an insufficiently high temperature for a reaction to take place.

Certainly, the consensus from Verschoor's (2011) experimental work as well as the archaeometric studies mentioned below is that the decoration occurred after firing. The medium by which the colour was fixed to the vessel surface is a matter for another investigation.

Turning now to a more archaeological conspectus, the heightened interest in an 'archaeology of the senses' in recent years in material culture studies has brought into sharp focus issues such as the presence and meaning of colour in prehistory, and particularly in the Neolithic (e.g. Jones and Bradley, 1999; Spence, 1999; Barber, 1999; Tacon, 1999; Boric, 2002; Jones and MacGregor, 2002; Hurcombe, 2007). Thus far there has been an emphasis on questions relating to colour symbolism and the visual effects of colour, but less work has been carried out on the physical properties of colour or the technological problems raised by the choice of colour during artefact production (Jones 2004, 334).

As mentioned above, colour is present at the Ness of Brodgar in the form of several stones from built structures which have designs placed on them through the use of colourant originating from mineral sources. The raw materials for the production of colour are also present on site, and show clear signs of having been worked (Card and Thomas 2012, 118-19). Small stone dishes also show evidence of colour, potentially from their use in preparing coloured pigment. Of further relevance is the possibility that some of the pottery from nearby Barnhouse may have been similarly decorated; Andrew Jones (pers. comm.), who examined the pottery from Barnhouse (Jones, 2005), was aware of this option but that at the time the evidence was not thought strong enough to warrant further work.

It is the presence of the white colourant manifesting itself as a thick white slip but also as a more fugitive layer on a number of sherds from Ness the site, identified as burnt bone ash, which is most surprising as this phenomenon has not been noted in the Scottish (or, indeed British) Neolithic hitherto. There are, however, some possible parallels, ranging from Beaker pottery in Scotland and Holland and third millennium BC Bell Beakers in Spain to Middle Bronze Age encrusted wares from Hungary.

D.L. Clarke was, perhaps, the first person to comment on the presence of a white paste incrustation in the comb impressions of some Scottish Beakers from Aberdeenshire (Clarke 1970, 10). He identified the relevant examples as being crushed burnt bone implying, however, that bone may have been used as a substitute due to the lack of accessible chalk deposits. His observations have been taken further by the Beakers and Bodies Project based in Marischal Museum, University of Aberdeen, which has recently examined 54 Beakers of which 31 had clear evidence of white paste (Curtis et al. 2010, 1). SEM-EDX analysis indicated the presence of calcium and phosphorus, and Raman spectroscopy demonstrated that this was in the form of calcium hydroxyapatite, the major inorganic element of bone. Curtis and his colleagues concluded that the paste appears to be ground-

down cremated bone, applied with an unidentified fixative. The dates gathered from ten skeletons associated with Aberdeenshire white paste Beakers range from 2470-2205 cal. BC to 2135-1935 cal. BC at 95.4% probability (*ibid*). Belonging to the same general period is a deep jar from Billown in the Isle of Man with a distinct colour scheme in red, white and black. Darvill and Andrews (2014, 535-56) proposed that its carbon-rich black band was achieved by rubbing an organic liquid onto the surface, perhaps immediately after firing when the organic matter would have carbonised and 'fixed' to the surface. Davis (2006) has reported deliberate carbon- blackening of the surface of some (EBA) decorated EBA Beakers and Pygmy Cups from contexts in Wales, while other examples of both beakers and cups had been decorated with a white inlay consisting of gypsum, calcite and calcined bone. A thick, often shiny, black coating on early Neolithic pottery from Carn Brea, Cornwall, was noted by Isobel Smith (Mercer et al. 1981, 170). Significantly, in two sherds the coating stopped short at a clearly demarcated line below the rim. Much of the pottery from the Sweet Track, Somerset, was described as having a deliberately applied black coating (Coles and Orme 1984, 44).

Further evidence for bone paste incrustation comes from recent research into the Dutch Funnel Beaker West Group (Trechterbekarcultur or TRB), dating between 3400 BC and 2850 BC (Verschoor 2011). Verschoor (2011) established that burned bone was involved in its white inlay. Her experimental work concluded that burned crushed bone was rubbed into fired pottery and then 'fixed' by the application of a fatty substance (such as bone marrow, glue or beeswax) which was shown to be resistant to taphonomic processes. In Spain, analyses of third millennium BC incrusted Bell Beakers from the Middle Guadiana river basin (inception *c*. 2800-2700 BC) have overturned the prevailing belief that the nature of the Bell Beaker white incrustation throughout the Iberian Peninsula was calcium carbonate (Odriozola and Hurtado Perez, 2007). Using a variety of analytical techniques (SEM-EDX, XRD and FTIR) it was shown that the incrustations were produced by the application of a thick, soft, moist paste produced by mixing dry powdered bone with a liquid, either water or a fatty agent (*ibid*). The literature on the Encrusted Pottery Culture of Middle Bronze Age Hungary (2000-1500BC) has commonly described the white inlays there as being composed of powdered shell or lime (Roberts et al., 2007). However, recent work using XRD and FTIR has shown that the most common material used to make inlays is mammal bone and that analysis of bone inlays using SEM revealed variation in the texture of inlays, perhaps related to the use of different skeletal elements and/or the age of the bone used (*ibid*). A combination of talc and hydroxyapatite (with minor instances of calcite, kaolinite and hydrocerussite) accounted for the white inlays in Neolithic-Bronze Age pottery from Piedmont in NW Italy (Giustetto et al., 2013).

It is generally accepted that the origin of Grooved Ware is in the far north of Scotland, and most probably in Orkney (Ashmore, 1998; Cleal, 1999; Jones, 2002; Sheridan, 2010; Thomas, 2010; Parker Pearson, 2012; Bayliss et al., 2017; Brophy and Sheridan 2012, 29) where the oldest, secure dates from well-stratified sites have been confirmed. There is still more to be done in narrowing down the date of its probable inception due to the problems created by the late fourth millennium calibration plateau, and the requirement for more secure dates (Sheridan 2010, 44).In this respect the ongoing work at the very large Ness of Brodgar site may prove to be of considerable value. However, the relatively rapid spread south of the Grooved Ware concepts, along with the creation of timber and stone circles and perhaps with other elements of a cultural package (Sheridan 2010, 33), shows that the flexibility and organic fluidity which appears to characterise much of inter-regional contacts in later Neolithic society may have been an important element in the spread of Grooved Ware. Clearly, the mobility of both people and ideas throughout the length and breadth of what is now Britain, and further afield, can be taken as established. In that context the new discoveries of bone paste decoration on pottery throughout Europe, from Orkney and Aberdeenshire in Scotland, and Holland in the north west to Italy, central Spain and Hungary, is perhaps less surprising than it might have been a few decades ago.

The dates above for the various locations of this phenomenon can be compared with the latest view that Grooved Ware in Orkney emerged between *c.* 3300/3200 and *c.* 3100 BC (Sheridan 2010:1). The inception of the Dutch Funnel Beaker West Group appears similar and the Scottish (Aberdeenshire), Spanish and Hungarian material may be somewhat later. Comparisons of this sort should, of course, be treated with caution. One interesting difference has, however, emerged. The Scottish, Dutch, Spanish and Hungarian examples all involved the application of bone paste as an inlay to incised decorative motifs on pottery, perhaps with an animal fat as a fixative. Incised Grooved Ware is a relatively small part of the assemblage from the Ness of Brodgar but, thus far, none has been identified as having a white inlay, with coloured decoration of all sorts being restricted to vessels with applied decoration. The exception to this is one sherd (SF 10930b) which has white colour applied to a horizontal groove on a plain applied cordon. This situation may change further with the on-going programme of analysis of the Ness of Brodgar assemblage.

Acknowledgements

RJ is indebted to Peter Chung for generous access to the SEM-Raman Facility in Earth Sciences, Glasgow University. He also thanks Effie Photos-Jones of Analytical Services for Art & Archaeology (Scotland) for SEM-EDX examination, Gert Petersen for thin section preparation, and the Chemistry Department at Glasgow for access to FTIR. Ingrid Mainland kindly provided cow bone from Ness of

Brodgar. RJ is grateful to Allan Hall for discussion of the XRD data, John Gilleece (Earth Sciences, Glasgow) for expertly preparing polished sections, and the Masters students who contributed to the first round of SEM-EDX and Raman work. The Ness of Brodgar Trust generously met the costs of the study reported here.

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584

SF analysis location	Si	Al	Fe	Ca	P	K	Ti	Mn	Zr	Sr	Rb
860 interior rim	22.2	7.7	2.7	0.5	1.3	2.3	4485	4282	nd	141	nd
860 exterior white rim	22.3	6.8	2.4	0.3	0.7	3.0	4630	514	nd	129	nd
861 fragment 1	22.9	7.8	1.0	0.3	0.9	2.3	4731	405	nd	177	nd
861 fragment 2	24.3	9.1	1.2	0.4	0.7	2.2	5167	759	nd	159	nd
882 body 1	22.9	5.8	2.8	0.7	0.5	2.2	5553	1059	nd	404	nd
882 body 2	18.7	4.1	2.2	0.5	0.3	1.7	4305	742	nd	192	nd
882 exterior white	19.8	7.1	3.0	1.2	3.4	3.6	4772	2115	nd	346	nd
882 exterior white 2	18.6	7.0	3.2	1.6	4.0	2.6	4901	2345	nd	340	nd
1518 body	17.6	4.9	3.2	0.4	0.5	2.4	3859	930	nd	161	nd
1518 ext no white	23.5	8.7	4.9	1.0	1.5	2.8	5278	1799	nd	296	nd
1518 ext white spot	16.5	5.7	3.9	2.7	6.1	3.1	4914	1128	nd	360	nd
6381 body	20.4	7.6	0.8	0.7	1.1	1.8	5284	411	nd	392	nd
6381 ext white	17.7	13.2	1.1	0.4	1.6	0.9	7738	161	nd	214	nd
6381 ext white 2	20.1	11.5	1.1	0.5	1.4	1.1	7895	303	nd	204	nd
6798 interior	17.8	5.1	3.96	0.9	1.2	2.07	5502	1031	154	290	67
6798 black	14.7	5.6	3.8	1.8	2.2	2.35	4302	713	129	218	60
8509 interior	10.2	5.25	3.4	0.9	0.74	2.02	4422	1220	200	315	76
8509 white 1	12.5	4.26	3.5	5.3	9.6	1.72	3012	2983	134	338	73

8509b interior	18.4	4.16	2.65	2	10	1.28	2642	1469	123	490	67
8509b white	15.7	4.4	3.9	2.4	8.5	2.27	3888	2079	148	550	53
9486 interior	15	3.35	1.5	5.4	1.6	1.31	2300	1071	79	452	50
9486 x section	16.1	4.5	5.9	1.4	1	1.66	5033	1718	104	1966	56
9486 x section2	15.7	4.6	5.8	2	0.6	1.37	6405	1733	116	530	39
9486 exterior band	19.3	7.8	4.6	1.6	2	3.42	4429	982	129	322	74
9871 black interior	9.4	2.2	2.6	3.6	1.6	1.16	2370	700	131	346	68
9871 x section	18.3	5.2	3.3	1.6	1.6	2.12	4735	1162	159	597	69
9871 white exterior	19.1	5.7	4	1.8	4.7	3.16	5375	1051	157	627	66
10101 interior	20.7	4.8	2.65	1.4	10.4	2.08	3611	1293	140	226	67
10101 red exterior	19	7.65	3.7	1.9	2	3.35	3584	1749	124	323	43
10252 interior	20.3	5.2	2.2	0.6	1.8	1.89	3899	334	198	191	65
10252 white exterior	19	4	2.1	1.3	3.35	1.79	3490	961	139	256	64
10533 interior	17.5	3.2	2.7	1.05	6.4	1.74	3399	924	153	334	35
10533 white exterior	16.2	4.1	2.4	2.8	3.5	2.12	3153	3767	152	263	39
10533 exterior no white	19.1	5.7	2.5	1.3	1.25	2.23	3988	1162	154	245	48
10541 interior	19.2	4.9	3.15	1.4	1.4	2.12	3956	1886	150	123	79
10541 black on raised band	17.8	6.4	3.05	0.9	1	2.08	4015	631	118	114	84
10930 interior	18.2	5.4	3.3	0.8	1	2.65	3280	2453	127	206	76

10930 reddish exterior	19.8	6.5	4	3	3.5	2.79	3261	1270	134	446	47
10930 least reddish area	19.5	6.35	4	2.1	2.8	3.94	3584	1007	126	337	48
10930b interior	21.4	5.85	3	2.1	1.8	1.88	3892	1410	159	337	69
10930b white exterior	13.6	4.5	2.8	6.5	8.7	1.91	2820	1458	114	691	31
10938 interior	7.8	2.6	2.9	1.1	1.3	0.86	3444	2352	136	535	60
10938 x section	10	2.2	3.2	1.5	10	1.26	2798	1316	115	243	59
10938 white in groove	6.3	2.25	1.7	1.3	12.4	1.15	1215	1173	104	441	55
10982 interior	22.1	5.9	3.8	2.7	2.3	1.87	4800	1283	150	392	44
10982 slip exterior	27.6	6.7	3.3	2.4	4	1.87	3410	8076	157	365	43
10982 slip exterior at edge	15.6	5.4	3.2	3	4.3	2.11	3101	5871	135	339	37
11247 interior	12.4	3.3	2.2	5.8	6	1.33	2610	882	126	251	59
11247 white exterior edge	16	4.9	2.6	4	7.9	2.1	3114	1153	149	354	59
11247 exterior unpainted	16.2	5.5	2.9	4.2	5.55	2.32	3231	1189	152	290	60
12018 interior	4.9	1.1	1.4	8.4	6.4	0.64	1240	810	116	268	49
12018 white exterior	17.8	4.5	2.3	4.3	4.2	2.21	3221	937	127	324	61
12018 exterior no white	22.8	6.4	2.6	1.9	1.7	2.4	3539	911	156	240	59

12631 white small	11.5	4.5	3.4	6.5	10.8	1.82	3451	3587	151	461	60
12631 large x section	16.4	4.7	3.8	2.85	0.86	1.58	4828	1589	153	477	57
14463 interior	20.3	4.8	2	2.3	1.8	1.72	3413	1084	144	272	56
14463 exterior1	9.9	2.06	2.1	7.7	9.6	1.59	2100	1530	127	427	45
14463 white in groove	16.7	4.3	2.3	3.7	3.9	2.08	3030	1647	138	375	49
14463 x section	14.8	3.4	2.85	1.2	1	1.9	2948	1553	137	256	57
27.437 body	17.0	5.4	3.3	0.9	1.2	1.6	4672	742	nd	227	nd
27.437 ext white	14.4	5.6	3.5	3.7	9.6	1.9	3455	3522	nd	342	nd

Table 2a The compositions of the decorated pottery determined by pXRF. Si, Al, Fe, Ca, P in %, the remainder in ppm. nd not determined; ext exterior; x section cross section.

SF & status	Ca	P	Si	Al	Fe	K	Ti	Mn	Sr
11458 carb	22.4	13.5	3.8	1.1	5300	2004	705	434	3743
11458 calc	12.4	7.9	16.5	4.6	17864	12055	2062	618	2076
11458 UB	11.4	6.7	6.2	1.6	10631	4077	593	4920	3861
12553 carb	31.8	15.9	3.3	1.2	3233	1244	411	319	4081
12553 UB	13.2	7.7	8.1	2.5	10339	5038	704	1142	4770
12553 calc	18.3	12.0	14.4	6.1	15261	8071	1145	2178	3524
8999 calc	28.9	19.3	2.0	1.3	5625	1355	611	719	2626
8999 UB	12.1	10.9	3.0	0.5	6580	1425	159	452	3980
8999 carb	21.0	16.6	1.6	<LoD	3901	1012	271	< LoD	4739
10818 UB	14.2	8.2	11.0	3.0	13577	6854	933	5149	4029
10817 calc	36.3	20.3	2.5	2.2	7614	1358	491	321	2207
14359 calc	40.3	21.4	1.6	1.8	4386	581	350	744	2580
10825 carb	12.0	8.3	12.1	3.2	17803	7649	1331	1778	2770
10825 calc	14.0	13.9	11.0	3.6	19874	9289	1809	4383	2210
11806 carb	21.1	12.5	5.0	1.6	4629	3265	590	363	3880
11806 calc	22.7	19.4	3.2	1.4	5922	2092	351	768	2281
11806 UB	17.7	13.3	2.4	0.8	4579	1649	384	576	3748

Table 2b The compositions of bone determined by pXRF. Ca, P, Si, Al in %, the remainder in ppm. Status of the bone: carb carbonised, calc calcined, UB unburnt. <LoD below limit of detection.